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Workshop

REPORT ON THE STANDARDIZATION WORKSHOP, 11th ICTAC 1996

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This was a very well attended meeting with the numbers reflecting an increasing world-wide demand for certified reference materials. This is an inevitable consequence of the growth of schemes for the accreditation of the quality of measurement.

Material standards for DSC and TG

ICTAC standards

The present situation with respect to ICTAC Standards was reviewed. These are available (through NIST) as sets but, because one or two components are out of stock, the whole operation is vulnerable. It is intended to change to the sale of individual materials to overcome this problem. It is recognised that there is a need for better temperature specifications (related to thermodynamic transition or melting temperatures) as well as the corresponding enthalpy changes. Ideally these should be obtained by independent methods (e.g. adiabatic calorimetry) but the cost in time alone rules this out for most potential standards. Instead, collaborative DSC work must be used for at least the majority of materials. Here it is essential to have some adiabatic (or other independent) data as reference markers for the DSC work.

Primary standards

Primary thermal standards (mainly metals but also some organics) are available from NIST, the UK Laboratory of the Government Chemist, and, soon, from the Physikalisch-Technische Bundesanstalt, Germany. In this respect 'DSC-certified' materials should be regarded as valuable secondary (i.e. working) standards in the chain of traceability to national/international standards. The conduct of the 'round

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robin' tests that lead to such secondary standards is a far from trivial operation. This is particularly relevant in the current climate where, increasingly, all time must be accountable. This does not encourage voluntary work – the source of all input to ICTAC programs – and we have the dilemma of fewer resources to meet an increased demand. Potential sources of funding to alleviate the problem were discussed.

DSC procedures and calibration in cooling

GEFTA, the German Thermal Analysis Society, has been particularly active in defining procedures to give data of quantitative significance and programs reported by E. Gmelin and S. M. Sarge appear elsewhere in this volume. GEFTA and other groups are also investigating potential standards for calibration in cooling. Most transitions require some degree of supercooling, the extent of which varies from sample to sample (this is easily seen in tin 'shot' where the solidification of individual particles may span several tens of degrees), certification is impossible. Fortunately, there is trivial supercooling (relative to the ± 0.1 K that is realistic for the best DSC methods) for many liquid crystal transitions and these were fully discussed. Care is still needed to ensure that the correct materials and procedures are used – low energy transitions, high purity, small samples (J. Thermal Anal., 49 (1997) 193.). It remained unclear if the 'onset' or 'peak' temperatures were appropriate for calibration purposes but the conditions given minimize the resultant uncertainties.

The glass transition

There has been an ICTAC standard for the glass transition temperature of polystyrene for many years. Our knowledge of the T_g region has improved greatly over this time (to a large extent, because of results obtained through the use of thermal methods) and better characterization is now needed. This will address the influence of thermal history in addition to the more usual instrumental variables – the two may be linked via the fictive temperature. There is a need for a corresponding inorganic glass standard and a potential material is available (through the courtesy of Professor Heide, Jena). Preliminary work has been successful and a more extended program of work will start soon.

Thermogravimetry

ICTAC standards for thermogravimetry are based on the magnetic transition temperature of a series of commercial alloys. Individual systems are calibrated by using simultaneous DSC-TG as described at the previous ICTAC meeting in Hatfield. Progress towards the tailoring of the transition temperature, based on Ni–Co and Ni–Pd systems, to any desired value from below ambient to 1121°C (cobalt) was described by Gallagher (this volume, p. 1013).

Although the above topics represented the interests of the majority of those attending the Workshop, there was a clear demand for standards for all thermal methods (in addition to the need for high temperature DSC calibrants and the new requirements of modulated DSC!). The effort that is needed is immense. It calls for YOUR involvement. If you feel that 'they' are not doing enough for your specific interests, remember that 'they', like you, have only limited time available. Please let us know your needs and your willingness to participate. SOME phase of ICTAC standards work will benefit from our mutual efforts.

Written standards for thermal analysis

Workshop time was specifically set aside to consider the many 'written' standards for thermal methods that are now appearing under the auspices of national standards bodies as well as ISO. The current situation in countries where there are groups active in promoting what are essentially 'standard operating procedures' was described:

1 Germany (DIN, Dr. S. Sarge, PTB)
2 Japan (JIS, Professor S. Nakamura, Kanagawa University)
3 USA (ASTM, Dr. R. Blaine, TA Instruments)
4 ISO work as reviewed by Pierre Le Parlouer (Setaram).

National standards have tended to precede those of ISO and there has already been considerable duplication of effort in production of the numerous very similar documents that now exist. In principle, national standards should give way to ISO documents when these become available but problems arise because standards may be developed on the basis of a technique or a material. Thermal methods lend themselves to the former approach, the specific needs of a particular class can then be addressed in the relevant material standard. For example, in determining the heat of cure of a resin or the melting of a polymer, both of which have lengthy baselines, it would be appropriate to specify the relevant (preferably ISO) standard and make some effort to define a correct baseline. As it is, existing procedures make no attempt to guide users to a sensible baseline and much of the care expended in the experimental approach is thrown away in the subsequent data treatment.